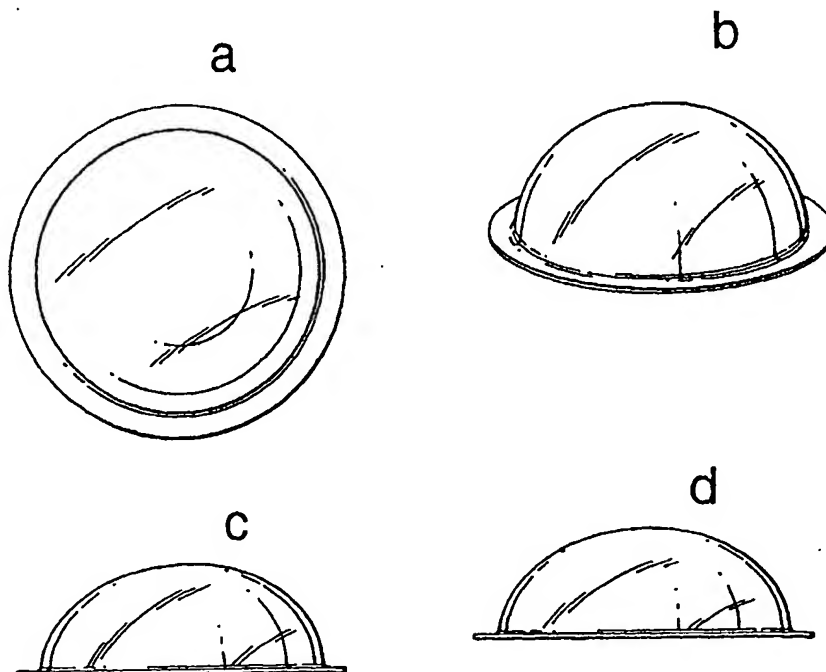




## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/EP00/01646 <b>(22) International Filing Date:</b> 29 February 2000 (29.02.00) <b>(30) Priority Data:</b> 9906175.6 17 March 1999 (17.03.99) GB <b>(71) Applicant (for AE AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT TZ UG ZA ZW only):</b> UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4P 4BQ (GB). <b>(71) Applicant (for all designated States except AE AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT TZ UG ZA ZW):</b> UNILEVER NV [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). <b>(71) Applicant (for IN only):</b> HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Maharashtra, 400 020 Mumbai (IN).		<b>(72) Inventors:</b> EDWARDS, David, Brian; 88 Hayfield, Chells Manor, Stevenage, Hertfordshire SG2 7JR (GB). McCARTHY, William, John; 1 Sunnyside Cottages, Donhead St. Mary, Shaftesbury, Dorset SP7 9DL (GB). RICHARDSON, Patricia; Lever Brothers Limited, 3 St. James's Road, Kingston-upon-Thames, Surrey KT1 2BA (GB). <b>(74) Agents:</b> ELLIOTT, Peter, William et al.; Unilever PLC, Patent Department, Colworth House, Sharnbrook, Bedford, Bedfordshire MK44 1LQ (GB). <b>(81) Designated States:</b> AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).  <b>Published</b> <i>With international search report.</i>

(54) Title: WATER SOLUBLE PACKAGE



## (57) Abstract

A water soluble package containing a fluid substance for release on dissolution of the package, the package having a dome shaped body portion for containing the fluid substance comprising a first sheet of a water soluble material thermoformed to form a body wall of the body portion, and a second sheet of water soluble material superposed on the first sheet and sealed thereto along a continuous region of the superposed sheets to form a base wall of the body portion.

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## WATER SOLUBLE PACKAGE

### INTRODUCTION

5 The invention relates to a water soluble package containing a fluid substance for release on dissolution of the package.

Detergent compositions for the machine washing of laundry are provided in many forms. Probably the most prevalent form  
10 of laundry detergent is washing powder or granules. A problem with the use of these forms of detergent is that the product needs to be dosed into the machine in such a way that the detergent is quickly and thoroughly dissolved in the wash water of the machine without coming into contact  
15 with the laundry in a solid form. In this regard many dosing devices which seek to overcome this problem have been proposed. One such device disclosed in European Patent Nos. 0 343 070 and 0 343 069 teaches the use of a flexible fabric sock which holds the particulate detergent in the  
20 machine, the fabric of the sock being permeable to water so as to allow water enter the sock and carry the detergent out of the sock through the fabric walls in the form of an aqueous solution. More recently unit dose forms of detergent have been proposed in the form of compressed tablets of  
25 detergent powder. A problem encountered with the provision of detergent tablets is that the tablets need to be strong enough to withstand storage and transport, yet weak enough to disintegrate and dissolve quickly in the washing machine. A further problem is the need to prevent the tablets  
30 "posting" in the porthole and between the drums of conventional washing machines. More recently these problems have been addressed by the provision of detergent tablets having specific chemical disintegrants which allow quick disintegration of the tablets in the aqueous environment of  
35 a washing machine, and by the provision of loosely fitting

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net bags which aid tablet disintegration and prevent "posting". However, as many of the current detergent tablets contain bleach and other irritant substances, the problem of handling the tablets remains.

5

The provision of detergent compositions in water-soluble films has been known for some time. Most of the documents relating to this subject describe water soluble film envelopes formed using a vertical form-fill-seal (VFFS) route. A problem with envelopes produced using this VFFS method is that, due to the constraints of the process, the resultant envelopes have seals which incorporate defined weak points where the seals overlap at corners. This results in envelopes which are easily corrupted as a result of impacts suffered during transport. In an attempt to overcome the problems associated with such VFFS envelopes, European Patent Application No. 0 608 910 describes thermoformed water soluble packages for pesticidal compositions. While this specification attempts to provide a solution to the problem of weak seals, the packages of EP-A-0608910 have other weak points and are designed specifically for containing pesticidal compositions.

The packaging and transport of water soluble packages containing fluid substances subjects the formed packages to considerable impact forces. A particular problem is that when a number of such packages are loose packed in a larger container which is then transported, the impact forces suffered by the packages within the container can be severe. The difficulty is that in such a situation it only takes one package in the larger container to break for the whole product to be ruined as far as the consumer is concerned because the fluid contents of the broken package may leak over any unbroken packages. Consumer confidence in a product is likely to be badly damaged by such an occurrence.

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The problem of minimising breakage to an acceptable level is particularly acute in the area of laundry detergents and other domestic consumer products and has not been solved until now..

5

It is an object of the invention to overcome at least some of the above disadvantages. It is a particular object of the invention to provide a water soluble package containing a fluid substance for release on dissolution of the package,  
10 which package has greater rupture resistance compared to known water-soluble packages.

#### STATEMENT OF INVENTION

15 According to the invention, there is provided a water soluble package containing a fluid substance for release on dissolution of the package, characterised in that the package has a body portion for containing the substance comprising a first sheet of a water soluble material  
20 thermoformed to form a body wall of the body portion, and a second sheet of water soluble material superposed on the first sheet and sealed thereto along a continuous region of the superposed sheets to form a base wall of the body portion, and in that the body portion of the package is  
25 generally dome shaped.

The applicants have surprisingly discovered that the above mentioned problems and disadvantages of prior art water soluble packages are substantially addressed by the packages  
30 according to the invention. In particular, the invention yields water soluble packages which are sufficiently robust to withstand (to a commercially acceptable level) the rigours of packaging and transport even when the fluid substance inside the package is a domestic consumer product  
35 such as a laundry detergent. The combination of

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thermoforming the packages of the invention and forming the packages into a dome shape confers surprising advantages on the packages of the invention. It is thought that thermoforming reduces stress on the film during forming  
5 compared to other forming techniques. As will be shown below, applicants have found that the dome is a shape which yields greater uniformity of film thickness over the package and greater impact resistance of the package.

10 In a preferred embodiment of the invention, the maximum height of the body wall above the base wall is preferably less than or equal to the maximum width of the base wall. The base wall is preferably generally circular but other dome shaped body portions according to the invention are  
15 envisaged. For example, dome shapes having rectangular, oval, square and triangular bases are envisaged. Preferably, the base of the dome will be substantially flat. Alternatively, the base may be somewhat concave or convex. In any case the resulting package is aysmmetrical about the  
20 base wall, although of course there can be one or more planes of symmetry perpendicular to the base wall.

The water soluble film, at least of the body wall, is thermoformable and, in one embodiment of the invention, is  
25 polyvinyl alcohol, or a polyvinyl alcohol derivative. Preferably the water soluble film of the base wall is the same material as that used to make the body wall. It is important that the body wall be thermoformed rather than cold formed because applicants have discovered that cold  
30 forming stresses the film and weakens the end package as a result.

Preferably, the thermoformed body wall of a package according to the invention is of substantially uniform  
35 thickness. By "substantially uniform" it is meant that at

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any measured point the thickness of the thermoformed film is preferably less than about  $\pm 25\%$ , even more preferably less than about  $\pm 20\%$  and most preferably less than about  $\pm 15\%$ , different from the original thickness of the film pre-thermoforming.

Preferably the film has a thickness of between 10 and 1000 microns. More preferably the film has a thickness of between 20 and 80 microns, most preferably between 40 and 60 microns.

In one embodiment of the invention, an exterior surface of the film is treated with BITREX<sup>TM</sup> to discourage ingestion of the package of the invention by children.

The substance contained within the package may be a liquid, a gel or a paste. If the substance is a liquid then preferably the liquid has a viscosity between 100 and 1000 centipoise, more preferably between 300 and 800 centipoise, even more preferably between 500 and 700 centipoise, and most preferably about 600 centipoise, when measured at 20°C at 105s<sup>-1</sup>. In a preferred embodiment of the invention the substance is present in an amount of between 10 and 500ml, preferably between 10 and 100ml, most preferably between 10 and 50ml. Suitably, the capsule contains between 20 and 30ml of a fluid composition. In a particularly preferred embodiment of the invention the fluid composition is a laundry treatment agent such as a laundry detergent, fabric conditioner or fabric care formulation. However, other compositions for domestic consumer use may be envisaged, such as disinfectants, personal care products, and the like. Preferably the composition is substantially non-aqueous, however the composition may comprise some water, for example between about 1 and about 5% water

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The invention also relates to a process for producing a package according to the invention, the process comprising the steps of:

- 5     • thermoforming a first sheet of a water soluble material to form a domed body wall of the body portion;
- placing the fluid substance in the body portion;
- 10    • superposing a second sheet of a water soluble material over the first sheet; and
- heat sealing the first and second sheets along a continuous region of the superposed sheets surrounding the
- 15    substance to form a base wall of the body portion.

In one preferred process the first sheet of water-soluble material is thermoformed by means of a heating plate. Preferably, the sheet of water-soluble material intimately

20   contacts the heating plate, typically by applying a vacuum between the heating plate and the sheet of water-soluble material. Generally, the vacuum applied will be of less than 0.6 Bar. Alternatively the sheet may be blown into contact with the heating plate. The thermoforming sheet can

25   be blown or sucked off the heating plate and into a suitable dome-shaped mould. In one aspect of the invention, the process includes an additional step of, prior to the addition of the fluid substance, applying a vacuum to the thermoformed body wall to maintain the shape of the body

30   portion at least until after the heat sealing step.

The invention also relates to a process for the machine washing of laundry by employing a package according to the invention, wherein the fluid substance contained within the



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package comprises a laundry treatment agent, the process comprising the steps of:

- placing at least one package into the machine along with  
5 the laundry to be washed; and
- carrying out a washing operation.

#### Detailed Description of the Invention

10

The packages of the invention are illustrated with reference to the drawings in which:

Figures 1a, 1b, 1c and 1d show different views of a first package according to the invention in which the base wall of  
15 the body portion is circular.

Figures 2a, 2b, 2c and 2d show different views of a second package according to the invention in which the base wall of the body portion is square.

Figures 3a, 3b, 3c and 3d show different views of a third  
20 package according to the invention in which the base wall of the body portion is oval.

Figures 4a, 4b, 4c and 4d show different views of a fourth package according to the invention in which the base wall of the body portion is triangular.

25 Figures 5a, 5b, 5c and 5d show different views of a fifth package according to the invention in which the base wall of the body portion is rectangular.

#### EXAMPLE

30

In this example a thermoforming process is described where a number of packages according to the invention are produced from two sheets of water soluble material. In this regard recesses are formed in the sheet using a forming die having  
35 a plurality of cavities with dimensions corresponding

- 8 -

generally to the dimensions of the packages to be produced. Further, a single heating plate is used for thermoforming the film for all the cavities, and in the same way a single sealing plate is described.

5

A first sheet of polyvinyl alcohol film is drawn over a forming die so that the film is placed over the plurality of forming cavities in the die. Each cavity is generally dome shape having a round edge, the edges of the cavities further  
10 being radiussed to remove any sharp edges which might damage the film during the forming or sealing steps of the process. Each cavity further includes a raised surrounding flange. In order to maximise package strength; the film is delivered to the forming die in a crease free form and with minimum  
15 tension. In the forming step, the film is heated to 100 to 120°C, preferably approximately 110°C, for up to 5 seconds, preferably approximately 700 micro seconds. A heating plate is used to heat the film, which plate is positioned to superpose the forming die. During this preheating step, a  
20 vacuum of 0.5 bar is pulled through the pre-heating plate to ensure intimate contact between the film and the pre-heating plate, this intimate contact ensuring that the film is heated evenly and uniformly (the extent of the vacuum is dependant of the thermoforming conditions and the type of  
25 film used, however in the present context a vacuum of less than 0.6 bar was found to be suitable) Non-uniform heating results in a formed package having weak spots. In addition to the vacuum, it is possible to blow air against the film to force it into intimate contact with the preheating plate.

30

The thermoformed film is moulded into the cavities blowing the film off the heating plate and/or by sucking the film into the cavities thus forming a plurality of recesses in the film which, once formed, are retained in their  
35 thermoformed orientation by the application of a vacuum

- 9 -

through the walls of the cavities. This vacuum is maintained at least until the packages are sealed. Once the recesses are formed and held in position by the vacuum, the composition, in this case a non-aqueous liquid detergent is added to each of the recesses. A second sheet of polyvinyl alcohol film is then superposed on the first sheet across the filled recesses and heat-sealed thereto using a sealing plate. In this case the heat sealing plate, which is generally flat, operates at a temperature of about 140 to 160°C, and contacts the films for 1 to 2 seconds and with a force of 8 to 30kg/cm<sup>2</sup>, preferably 10 to 20kg/cm<sup>2</sup>. The raised flanges surrounding each cavity ensure that the films are sealed together along the flange to form a continuous seal. The radiussed edge of each cavity is at least partly formed by a resiliently deformable material, such as for example silicone rubber. This results in reduced force being applied at the inner edge of the sealing flange to avoid heat/pressure damage to the film.

Once sealed, the packages formed are separated from the web of sheet film using cutting means. At this stage it is possible to release the vacuum on the die, and eject the formed packages from the forming die. In this way the packages are formed, filled and sealed while nesting in the forming die. In addition they may be cut while in the forming die as well.

During the forming, filling and sealing steps of the process, the relative humidity of the atmosphere is controlled to ca. 50% humidity. This is done to maintain the heat sealing characteristics of the film. When handling thinner films, it may be necessary to reduce the relative humidity to ensure that the films have a relatively low degree of plasticisation and are therefore stiffer and easier to handle.

- 10 -

**EXPERIMENTAL DETERMINATION OF OPTIMUM PACKAGE SHAPE****i) Film Thinning During Thermoforming**

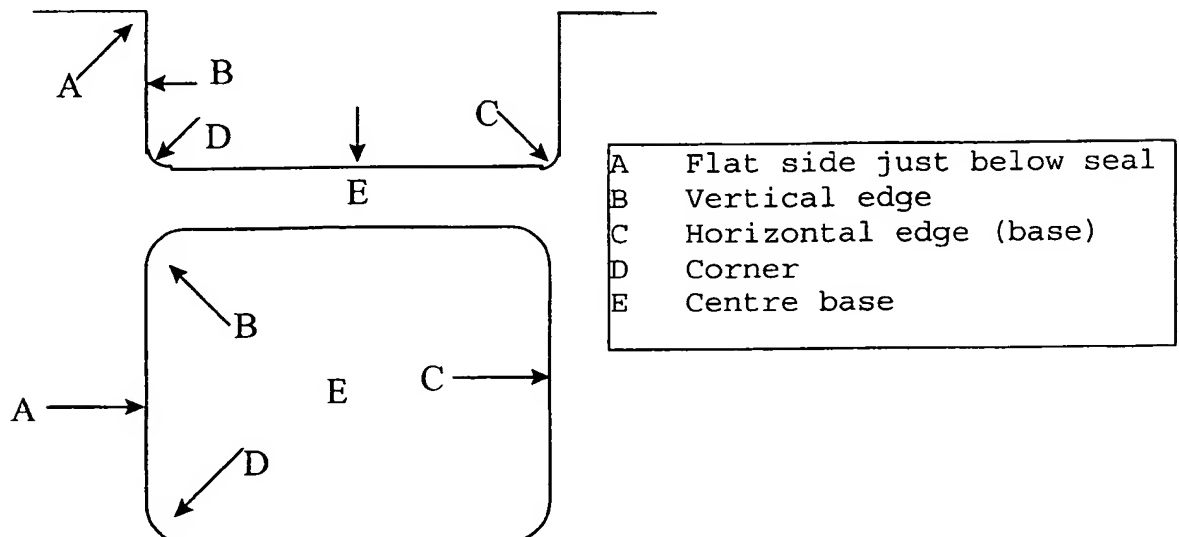
- 5 The objective of this experimental work was to create thermoformed packages different shapes from the same material under the same conditions with the same depth of draw and approximately the same volume.
- 10 The formed packages were then tested for thickness at specific points using a micrometer. The results were then compared between shapes,

The conditions used were:

- 15 Film: Polyvinyl alcohol (supplied  
By Chris Craft under  
Reference CC8534)
- Thickness: 75 $\mu$ m
- 20 Volume of cavity: Approx. 30ml
- Atmospheric condition: 17°C, 46% RH
- Micrometer sensitivity: 5 $\mu$ m

**Measurement points**

25

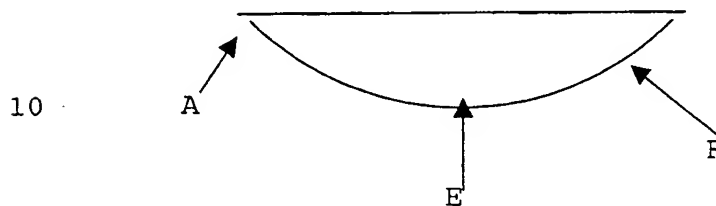


- 11 -

The rectangular shape shown is used only as an example to show the various points.

For a dome shaped package only points A & E are the same.

- 5 Points B, C and D are all equivalent in the dome shape and they were represented by Point F below.



The results were as follows:

15

Shape	Sample	Draw Depth	Measurement in microns					
			A	B	C	D	E	F
Dome	1	20mm	60				70	65
	2	"	65				60	65
	3	"	65				60	60
Triangle	1	"	50	50	45	30	65	
	2	"	55	55	40	25	55	
	3	"	55	55	40	25	60	
Dome	4	25mm	60				70	70
	5	"	65				70	70
	6	"	65				65	70
Cube	1	"	55	40	20	15	35	
	2	"	65	35	25	20	35	
	3	"	60	40	25	20	40	

These results show that while thinning always takes place on thermoforming:

20

- a) for the dome shape thinning is uniform over the test points; and
- b) the dome has a lower level of thinning than the others over the test points.

25

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ii) Impact Testing

The objective of this test was to show any differences,  
which exist, between the impact resistance of the dome shape  
5 and other shapes.

A falling dart impact test was used.

The test specimen was placed on a hard surface immediately  
10 below the suspended dart. The dart was allowed to fall,  
striking the specimen centrally. The specimen was then  
examined and any rupture or leak recorded. If there was no  
rupture or leak, the specimen was subjected to successive  
impacts, each at a higher dart weight, until rupture did  
15 occur.

Three shapes of package were used, all of the same material,  
fill and approximate weight and size. The test was repeated  
on both sides of the package (base side up and base side  
20 down) and several replicates were tested and a mean taken.

Before testing all specimens were exposed to the conditions  
of the test site for 12 hours to reach equilibrium. Test  
details were:

25

Dart: smooth hemispherical impact surface of  
38mm diameter

Drop height: 615mm

Surface: Smooth non-resilient plate

30 Conditions: 20°C, 70% RH

Dome, triangle (ie triangular pyramid) and cube shapes were  
tested.

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Each package was formed from polyvinyl alcohol supplied by Chris Craft under reference CC8534. The film thickness was 75µm.

5 Results were as follows:

Sample	Shape	Orientation	No. of Drops	Dart Weight to Rupture	Observations
1	Dome	Base Up	3	250g	Small hole mid base
2	Triangle	"	2	150g	2 holes in edges
3	Cube	"	2	150g	3 or more holes in corners & edge
4	Dome	Base Down	4	350g	hole in base

The above results show that the impact resistance of dome is  
10 greater than the cube or triangle.

### iii) Secondary Packaging Test

This experimental test was designed to establish any  
difference in impact survival between dome shaped packages  
15 and triangular packages of a liquid detergent product when  
multiple packages are contained in fibreboard box

22 dome-shaped packages according to the invention,  
containing a liquid laundry detergent, were placed randomly  
20 in a fibreboard box. The box was made of "M-flute"  
material of dimensions 170 x 85 x 54 mm which had a total  
filled weight of 609g.

The box was sealed closed and subjected to a vertical drop  
25 of 1.2 m on to a hard flat surface on the short dimensions.

This test was repeated for triangular packages of the same  
weight and film.

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The results of the test were that 3 out of the 22 dome shaped packages were found to have a small amount of leakage but with the triangular packages, 7 of the 22 were leaking  
5 and in 5 cases leakage was very severe.



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**CLAIMS**

1. A water soluble package containing a fluid substance for  
5 release on dissolution of the package, characterised in  
that the package has a body portion for containing the  
substance comprising a first sheet of a water soluble  
material thermoformed to form a body wall of the body  
10 portion, and a second sheet of water soluble material  
superposed on the first sheet and sealed thereto along a  
continuous region of the superposed sheets to form a  
base wall of the body portion, and in that the body  
portion of the package is generally dome shaped.
- 15 2. A package according to claim 1, characterised in that  
the maximum height of the body wall above the base wall  
is less than or equal to the maximum width of the base  
wall.
- 20 3. A package according to claim 1 or claim 2, characterised  
in that the base wall is generally circular.
4. A capsule according to claim 1 or claim 2, characterised  
in that the base wall is generally oval.
- 25 5. A package according to any one of claims 1 to 4,  
characterised in that the base wall is generally flat,  
or slightly concave or convex.
- 30 6. A package according to any one of claims 1 to 5,  
characterised in that the first sheet of water-soluble  
film comprises polyvinyl alcohol or a polyvinyl alcohol  
derivative.

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7. A package according to any one of claims 1 to 6,  
characterised in that the second sheet of water soluble  
material comprises polyvinyl alcohol or a polyvinyl  
alcohol derivative.
- 5 8. A package according to any one of claims 1 to 7,  
characterised in that the fluid substance is a liquid,  
paste or a gel.
- 10 9. A package according to any one of claims 1 to 8,  
characterised in that the fluid substance is a domestic  
consumer product.
- 15 10. A package according to claim 9, characterised in that  
the substance is a laundry detergent composition.
11. A process for producing a water soluble package having a  
body portion containing a fluid substance for release on  
20 dissolution of the package, characterised in that the  
process comprises the steps of:
- thermoforming a first sheet of a water soluble material to  
form a domed body wall of the body portion;

25

  - placing the fluid substance in the body portion;
  
  - superposing a second sheet of a water soluble material  
over the first sheet; and

30

  - heat sealing the first and second sheets along a  
continuous region of the superposed sheets surrounding the  
substance to form a base wall of the body portion.

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12. A process according to claim 11, characterised in that the first sheet of water-soluble material is thermoformed by means of a heating plate.

5

13. A process according to claim 11 or claim 12, characterised in that prior to the addition of the substance a vacuum is applied to the thermoformed body wall to maintain the shape of the body portion at least until after the heat sealing step.

10

14. A process for the machine washing of laundry by employing a package according to any of claims 1 to 10, characterised in that the fluid substance contained within the package comprises a laundry treatment agent, the process comprising the steps of:

15

- placing at least one package into the machine along with the laundry to be washed; and

20

- carrying out a washing operation.

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Fig.1a.

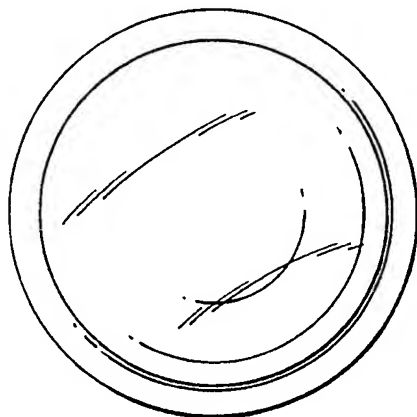


Fig.1b.

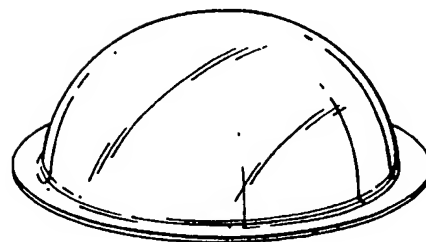


Fig.1c.



Fig.1d.



Fig.2a.

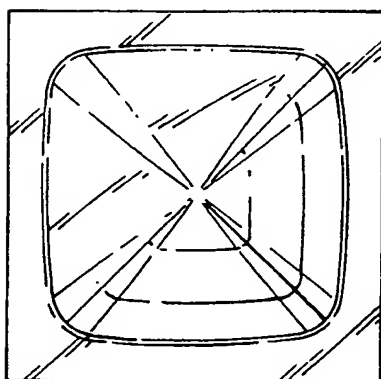


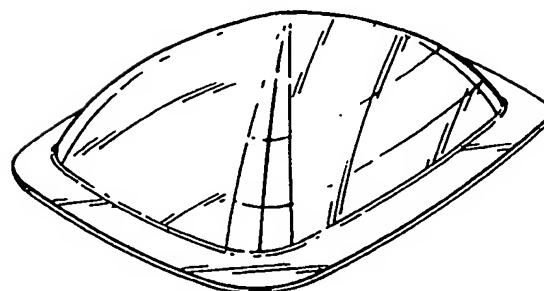
Fig.2b.



Fig.2c.



Fig.2d.



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Fig.3a.

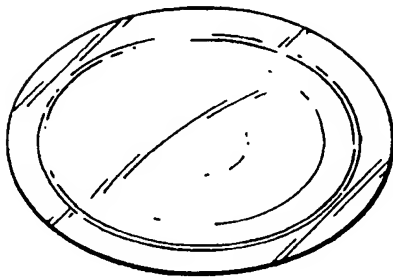


Fig.3b.



Fig.3c.



Fig.3d.

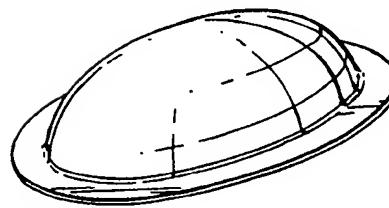


Fig.4a.

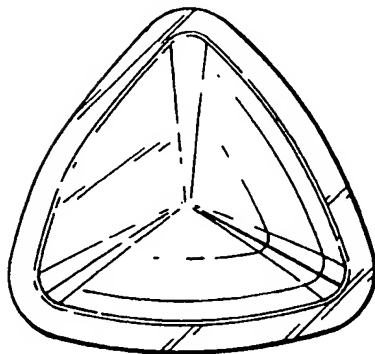


Fig.4b.

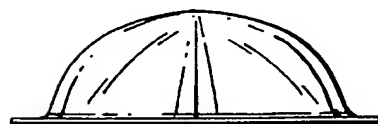


Fig.4c.



Fig.4d.

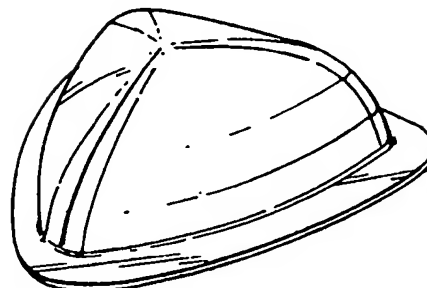


Fig.5a.

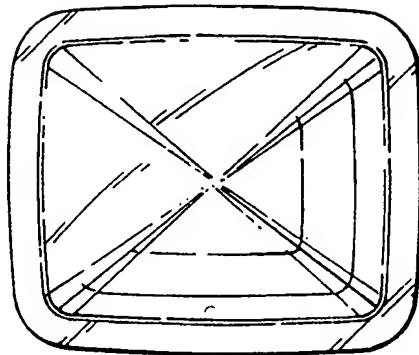


Fig.5b.



Fig.5d.

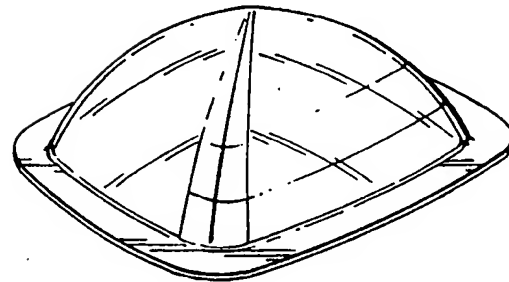


Fig.5c.



## INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/01646

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 B65D65/46

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 B65D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	WO 89 04282 A (KOSKA & WATTS LTD ;MARKBEECH PACKAGING (GB)) 18 May 1989 (1989-05-18) page 1, line 1 - line 6 page 9, line 22 -page 10, line 7; figures 1,2	1-12,14
A	US 2 377 118 A (WEISMAN) 29 May 1945 (1945-05-29) page 2, line 37 - line 50; figures 6,7  -/--	1

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

22 May 2000

Date of mailing of the international search report

29/05/2000

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# INTERNATIONAL SEARCH REPORT

Inter- national Application No

PCT/EP 00/01646

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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A	EP 0 366 231 A (ZIMPRO PASSAVANT INC) 2 May 1990 (1990-05-02) column 4, line 5 - line 10; claims 1,11; figure 1 -----	1,13
A	US 4 973 416 A (KENNEDY SHAUN P) 27 November 1990 (1990-11-27) claims 1,2 -----	1



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